

**United States Department of Agriculture
Agricultural Marketing Service, Science & Technology
Pesticide Data Program**

SOP No.: PDP-LABOP-12		Page 1 of 10
Title: Poultry and Beef Adipose, Liver, and Muscle Analysis		
Revision: 2	Replaces: 10/01/00	Effective: 04/01/01

1. Purpose:

To provide standard procedures for the receipt, preparation, storage, analysis, reporting, and disposal of USDA, AMS Pesticide Data Program (PDP) poultry and beef samples.

2. Scope:

This standard operating procedure (SOP) shall be followed by the analytical laboratory conducting pesticide residue studies for poultry and beef, the USDA, AMS Eastern Laboratory (US2). Sampling shall be performed by designated USDA, Food Safety and Inspection Service (FSIS) inspectors under established sampling protocols. All samples shall be shipped frozen to the USDA, AMS Eastern Laboratory. Each sample shall be comprised of adipose, liver, and muscle tissues. Any reportable residue results shall initiate immediate action reporting to the USDA, AMS Pesticide Data Branch and the official USDA, FSIS designee.

3. Outline of Procedures:

- 6.1 Sample Receipt
- 6.2 Sample Preparation
- 6.3 Sample Storage
- 6.4 Sample Analysis
- 6.5 Data Reporting
- 6.6 Sample Disposal

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4. References:

- Attachment 1, FSIS Unified Sampling Form, Form 10,210-3 , effective January 1, 1998
- Attachment 2a, PDP Sample Information Form for Poultry, effective January 1, 2000
- Attachment 2b, PDP Sample Information Form for Beef, effective April 1, 2001
- Attachment 3a, PDP Sampling Site Code List for Poultry
- Attachment 3b, PDP Sampling Site Code List for Beef
- Attachment 4a, PDP Poultry Tolerance Reference Table, effective April 1, 2000
- Attachment 4b, PDP Beef Tolerance Reference Table, effective April 1, 2001
- Attachment 5, Reportable Residue Form for Poultry and Beef, effective April 1, 2001
- Memorandum, Martha Lamont, PDP Technical Director, to Ed Zager, Chief, EPA/HED, August 24, 2000
- USDA/AMS, USDA OPMP, EPA Planning Meeting, August 23, 2000
- Memorandum, OPs in Meat and Poultry, Martha Lamont, EPA/HED, June 8, 1998

5. Summary:

No site shall be sampled more than once on a single day. Each poultry sample shall be a frozen composite of six birds from the same flock and each beef sample shall be collected from a single animal. Each sample shall be comprised of approximately one pound each of adipose, liver, and muscle. Each tissue shall be analyzed for identified compounds (refer to SOP PDP-QC-13J, poultry, or SOP PDP-QC-13P, beef). Any results $\geq 80\%$ of the established tolerance, or present where no tolerance is established, shall be reported immediately to the USDA, AMS PDB and USDA, FSIS using the PDP Reportable Residue Form for Poultry and Beef (see attachment 5).

6. Procedure:

This SOP represents minimum PDP requirements for the receipt, preparation, storage, analysis, reporting, and disposal of USDA, AMS PDP poultry and beef samples and is presented as a general guideline. The analytical laboratory shall have written procedures that

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provide specific details concerning how the procedure has been implemented in the laboratory.

6.1 Sample Receipt

- a. Quarterly sampling plans designating sampling dates and sites shall be prepared by USDA, FSIS and provided to the analytical laboratory. Samples shall be collected on Mondays, except when Monday is a holiday. In this case, samples shall be collected on Tuesday. Samples shall be frozen overnight and shipped to the laboratory by overnight courier no later than two days after the sample is collected. If a sample is not received during the expected timeframe, the laboratory shall immediately contact the PDP Sampling Manager and the designated USDA, FSIS contact, Dr. Martha E. Styles, at (phone) 402-221-7401, extension 7453.
 - b. Each poultry sample sent shall represent a composite of tissue from six birds in the same flock and each beef sample shall be collected from a single animal. Each sample shall be comprised of approximately one pound each of adipose, liver, and muscle tissue.
 - c. Samples shall be shipped frozen. Samples that have not completely thawed in transit (still cold to the touch) may be homogenized and stored until time of analysis. Samples and homogenates shall be held in the freezer at 0°C or lower until analysis.
 - d. Those samples, or portions thereof, received in a damaged (e.g., warm to the touch, spoiled, or leaking) condition shall be discarded and not analyzed. Condition and disposal shall be recorded on all applicable documentation, for example, the appropriate USDA, FSIS Sampling Form 10,210-3 and PDP Sample Information Form (SIF). If a sample must be discarded, the laboratory shall immediately notify the USDA, AMS, PDB and Dr. Martha E. Styles, USDA, FSIS, at (phone) 402-221-7401, extension 7453, or (fax) 202-221-7497.
 - e. Each sample shall be accompanied by a Unified Sampling Form (see
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attachment 1) completed by the USDA, FSIS inspector collecting the sample. The laboratory shall assign the PDP sample identification number. This number shall be a concatenation of the state, sampling date, sampling site, tissue, and analytical laboratory. This information is located in the following blocks of the USDA, FSIS Unified Sampling Form: state (6), sampling date (19), and sampling site (3).

NOTE: The USDA, FSIS site code is a seven-digit code which must be cross-referenced with the PDP Site Code List (see attachment 3).

Poultry and beef tissues shall have the following commodity codes, respectively: adipose (PA, BA), liver (PL, BL), and muscle (PM, BM). The laboratory designation is US2.

Example: For a poultry sample collected at site 3002 in Wilkesboro, North Carolina, on April 15, 2000, the following sample identification would be entered on the appropriate PDP SIF:

adipose (NC-00-04-15-3002-PA-US2)

liver (NC-00-04-15-3002-PL-US2)

muscle (NC-00-04-15-3002-PM-US2)

- f. The laboratory shall maintain a log of samples received. Refer to SOP PDP-LABOP-01, subsection 5.1.

6.2 Sample Preparation

Samples may be homogenized prior to extraction and stored in the freezer at 0°C or lower. Mechanically homogenize each tissue until a visually homogeneous mixture is attained. The laboratory shall use its discretion in the utilization of dry ice during the homogenization procedure.

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6.3 Sample Storage

An adequate portion of the homogenized tissue shall be held in reserve in case re-analysis is required. This portion shall be distributed among several small containers (polypropylene or styrofoam recommended) rather than one large container and stored at 0°C or less. The laboratory's internal documentation shall specify or define an "adequate portion" and distribution.

6.4 Sample Analysis

a. Weighing of Analytical Portion

An appropriate amount of homogenized tissue shall be weighed for analysis. The laboratory internal SOP shall define the sample weight and the necessary precision which, for a 10 gram sample, shall not be more than +/- 0.05 grams, and for a 20 gram sample, shall not be more than +/- 0.10 grams.

b. Sample Set Requirements

A sample set is the group of test portions that are spiked with the designated process control and extracted on a single day along with the required QC samples. Each set shall consist of no more than 20 analytical samples. Required QC samples per set include a reagent blank, matrix blank, and matrix spike(s).

The matrix spike(s) shall be prepared at approximately 2xLOQ with the identified marker pesticides. Refer to SOP PDP-QC-13J for poultry and SOP PDP-QC-13P for beef.

Each analytical sample shall be spiked with the appropriate process controls at approximately 5xLOQ. All components of sample sets shall be subject to the entire analytical test process as detailed in approved methodology.

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c. Analytical Requirements

Each tissue shall be analyzed for compounds as specified in SOP PDP-QC-13J, poultry, or SOP PDP-QC-13P, beef.

The designated process control for the HPLC/post-column system is isoprocarb. The designated process control for GC instruments is ronnel (fenchlorphos).

Some analytical reference materials are not readily available from commercial sources, therefore, materials may be requested from the US EPA Standards Repository. Please contact Dick Griffith, EPA, Fort Meade, MD, at 410-305-2905 (phone) or 410-305-2999 (fax) or Chuck Stafford at 410-305-2914. Other compounds may also be requested from the EPA Standards Repository, if desired.

NOTE: Use of alternate suppliers whose reference material fails to meet the minimum requirements outlined in SOP No.: PDP-STD-01 must be approved by the PDP Technical Director and documented.

d. Method Validation Requirements:

All compounds shall undergo validation requirements according to SOP PDP-QC-07. Completion of the following validation/evaluation modules is required for each new tissue /compound:

- 7.3 establish LODs
- 7.4 verify LODs
- 7.5 determine consistent instrument response
- 7.6 determine method performance

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The following modules may be performed concurrently with sample analysis:

- 7.7 precision and accuracy (seven data points for each compound)
- 7.8 final results reporting

6.5 Data Reporting

a. Reportable Residues

If a sample contains $\geq 80\%$ of the established tolerance, or contains a residue and no tolerance is established, then a reportable residue has occurred. The Reportable Residue Form for Poultry and Beef (attachment 5) shall be filled out and faxed immediately to the USDA, AMS, PDB and USDA, FSIS. The USDA, FSIS contact is Dr. Martha E. Styles:

Dr. Martha E. Styles (fax: 202-221-7497)
FSIS Technical Services Center
1299 Farnam Street, Suite 300
Landmark Center
Omaha, Nebraska 68102

b. Presumptive Tolerance Violations (PTVs)

Reporting requirements, as specified in SOP PDP-DATA-02, subsection 5.3 do not apply.

c. All data (manual or electronic) shall be transmitted according to established procedures.

6.6 Sample Disposal

Stored homogenates shall be disposed of when all requirements for acceptability criteria have been met and results have been successfully transmitted to USDA, AMS, PDB. Disposal shall be documented (e.g., freezer log, sample log, extraction worksheet, RDE chain-of-custody inputs)

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and shall contain a minimum of date of disposal, sample number, and initials of the individual who discarded the sample.

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3/27/01

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- Added provision for beef analytical procedures

